Ultra High-Resolution X-ray Topography using a Third-Generation Synchrotron Light Source

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Stony Brook Synchrotron Topography Facility Beamline X-19C

National Synchrotron Light Source

STONY BROOK STATE UNIVERSITY OF NEW YORK

Department of Materials Science & Engineering

Preview for X-ray topography at NSLS-II

Science theme

- defect and strain studies of wide range of single crystal materials (metals, organic materials, semiconductors, optical materials) -> improve their crystalline quality

What can we gain from NSLS-II?

- **Ultra high spatial resolving power for defects (dislocations)**

currently no technique can adequately cover the gap in resolving power (10^6 – 10^8/cm^2) between XRT (0 – 10^6/cm^2) and TEM (10^8 -).

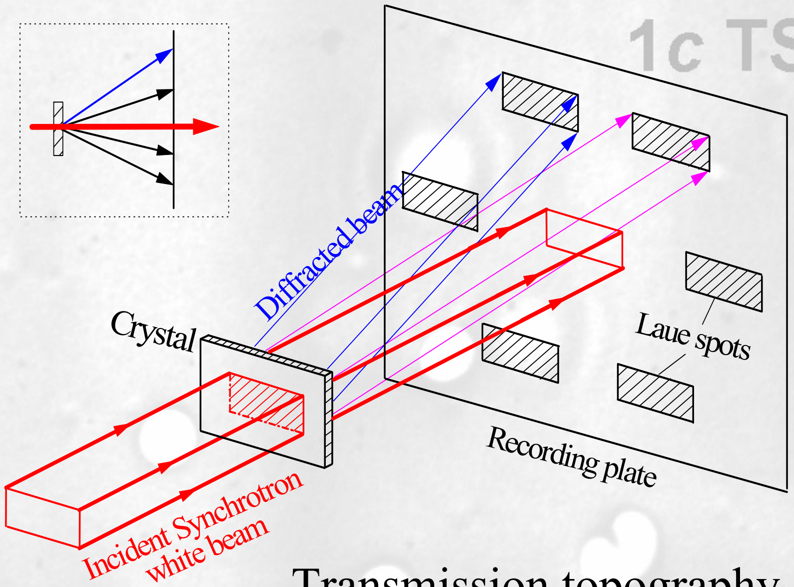
-Ability to detect very small strains distributed over large areas

Utilization in industry and science community

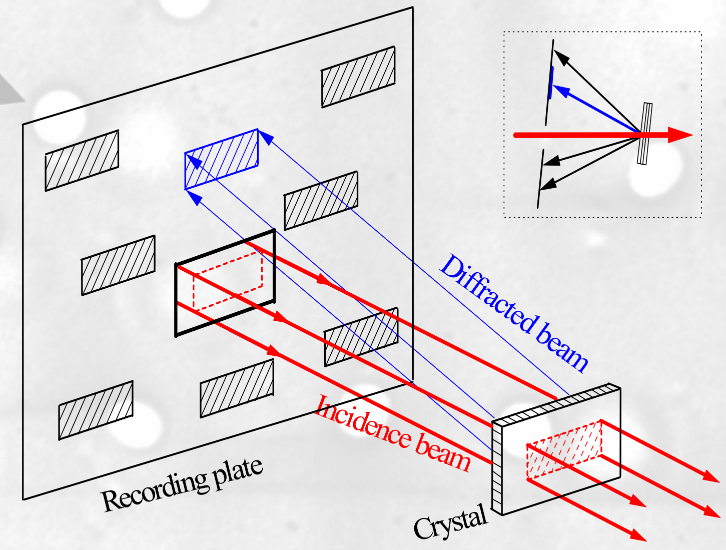
the improved resolution provides great opportunity for industry and research community to study defects in various crystals particularly wide bandgap semiconductor such as SiC (defect density 10^4 – 10^6/cm^2) and GaN, AlN and related alloys (defect density 10^6 – 10^{10}/cm^2)

Qualification of single crystal optical elements (polishing damage, defects, strains)

Synchrotron X-ray topography

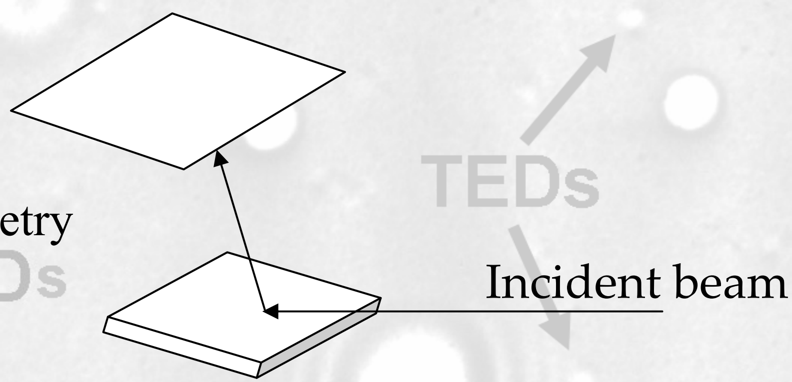


Transmission topography
Laue geometry



Back-reflection geometry

Grazing-incidence geometry



Comparison between white beam and monochromatic

White beam topography

Advantage:

- wavelength satisfying Bragg condition is automatically selected
- multiple reflections can be recorded simultaneously

Disadvantages:

- High signal/noise ratio
- limited specimen-to-film distance due to background. Typically must be greater than around 5cm (this limits resolving power).

Monochromatic topography

Advantage:

- higher strain sensitivity
- much lower noise
- specimen-to-film distance can be extremely small (<1 cm)

Disadvantages:

- sample alignment is more time consuming

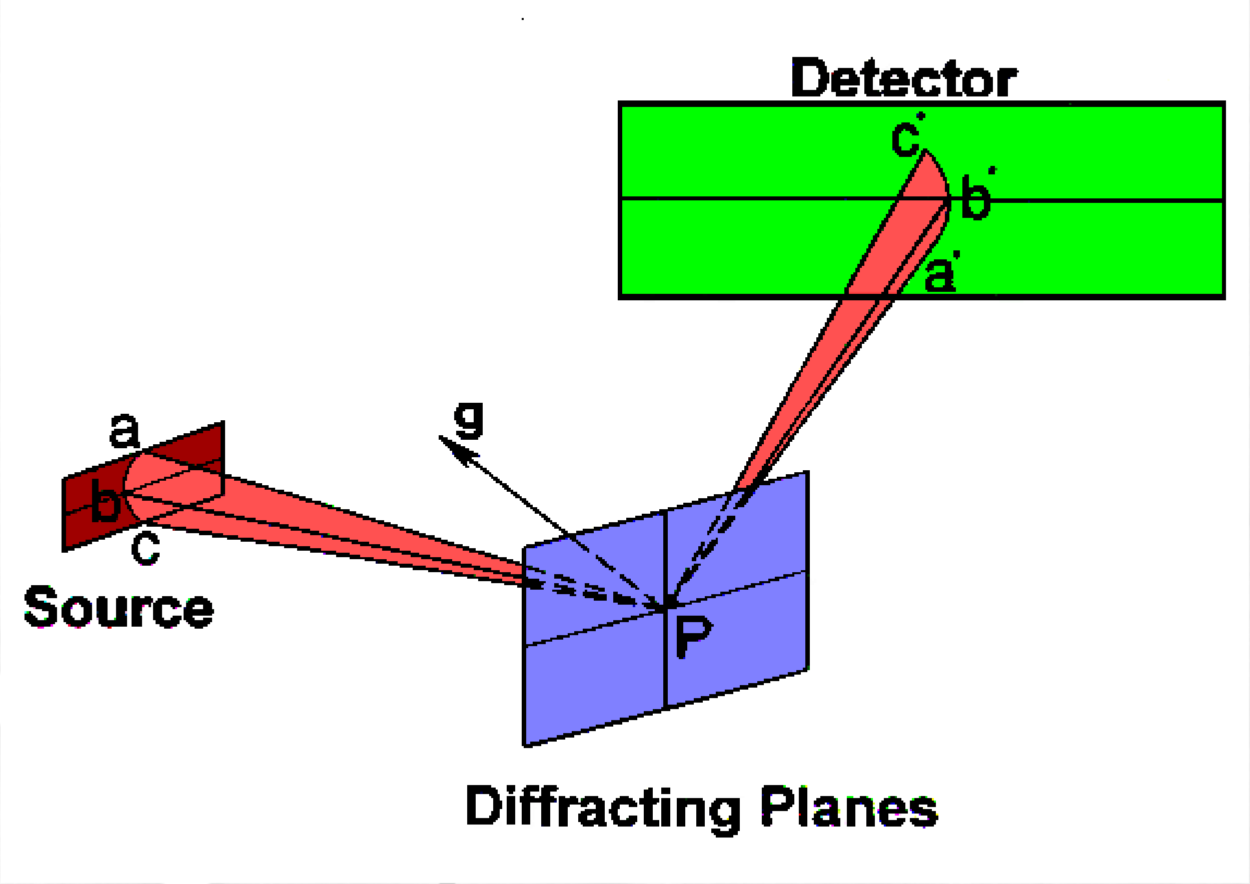
Theoretical spatial resolution of x-ray topography

$$R = a'c' = \frac{(ac)(Pb')}{bP}$$

ac: source size;

Pb′: specimen-to-film distance

bP: source-to-specimen distance

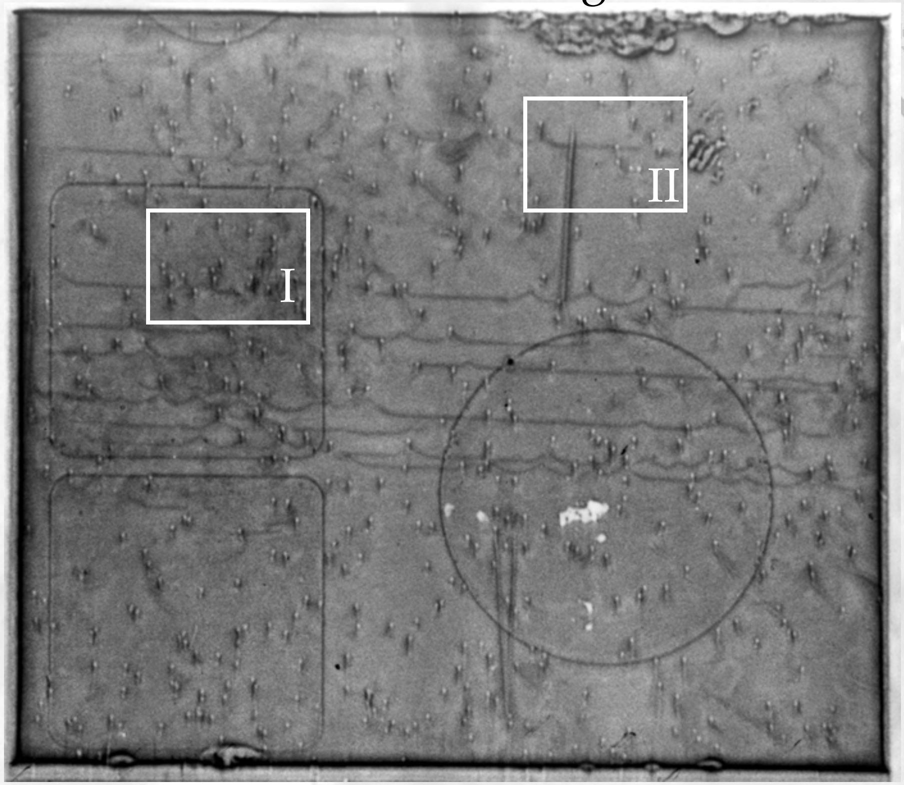


To obtain higher resolution:

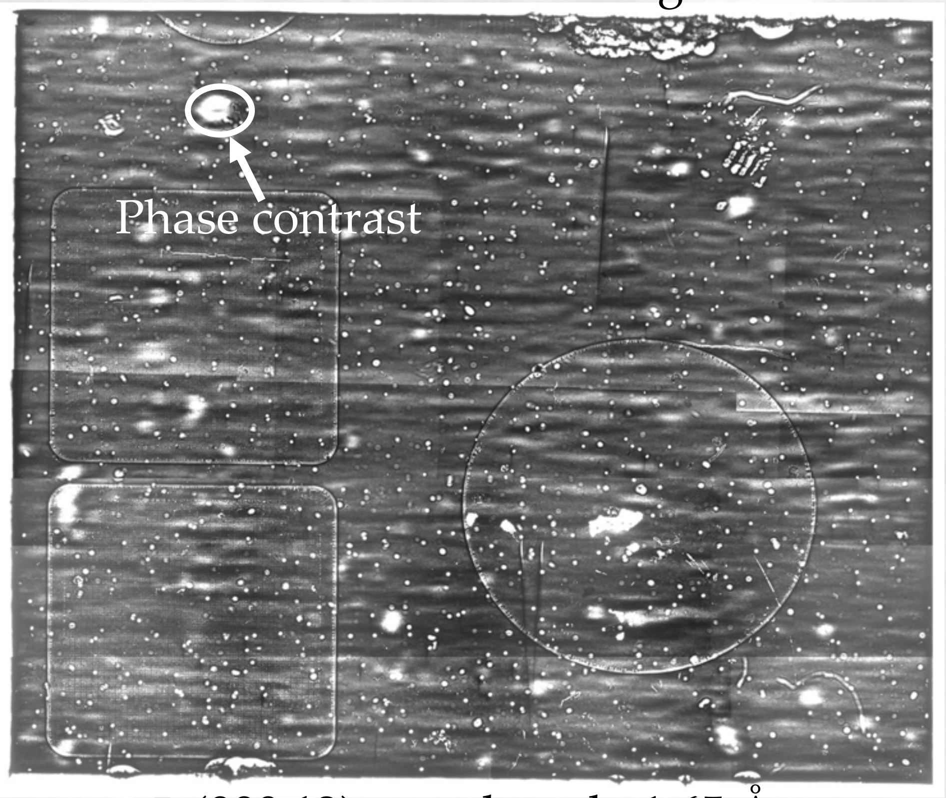
1) White beam vs. monochromatic beam? 2) high-resolution detector; 3) Reduce the source size; 4) increase the source-to-specimen distance; 5) reduce specimen-to-film distance

Comparison between white beam and monochromaticC

White beam image



Monochromatic image

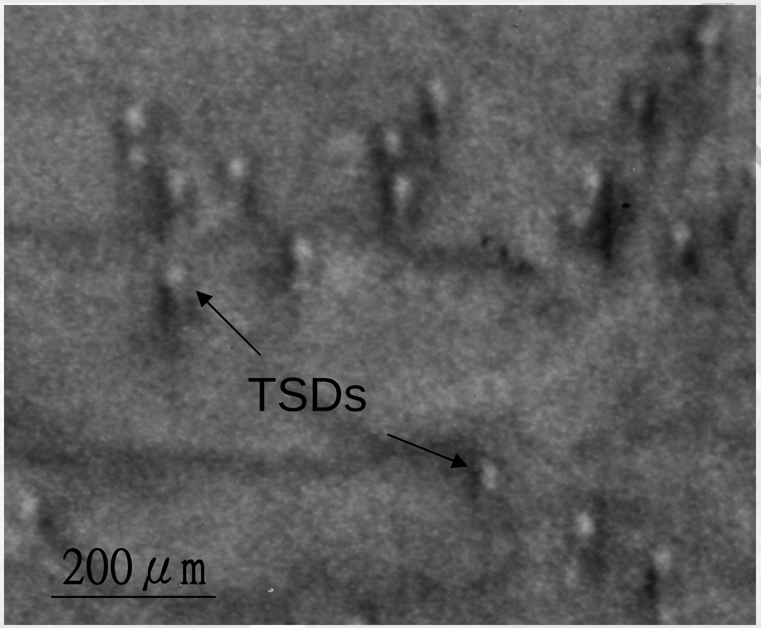


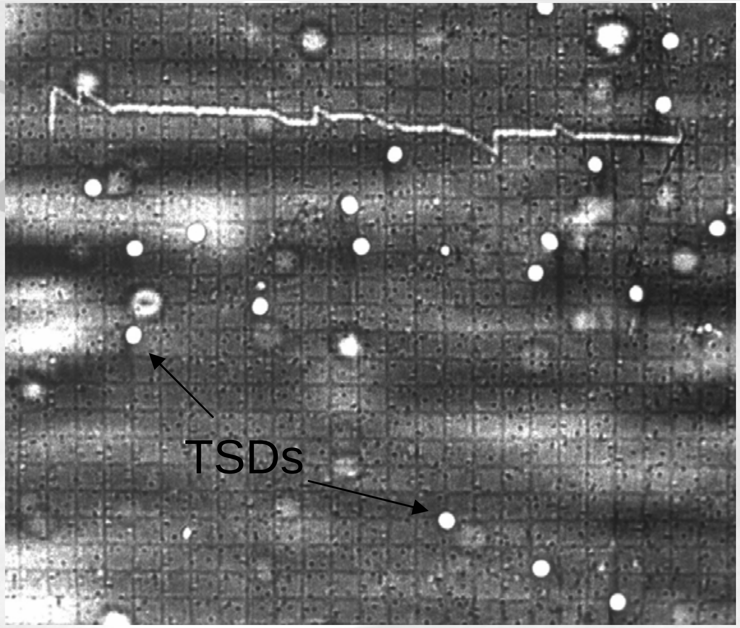
(000.12) wavelength=1.65 Å
Penetration depth: 28 μm

	Diffracted intensity(%)	Penetration depth(μm)
(00012) 1.65Å	10%	28.7
(00016) 1.24Å	45%	64
(00020) 0.99Å	27%	122
(00024) 0.83Å	10%	212
(00028) 0.71Å	8%	340

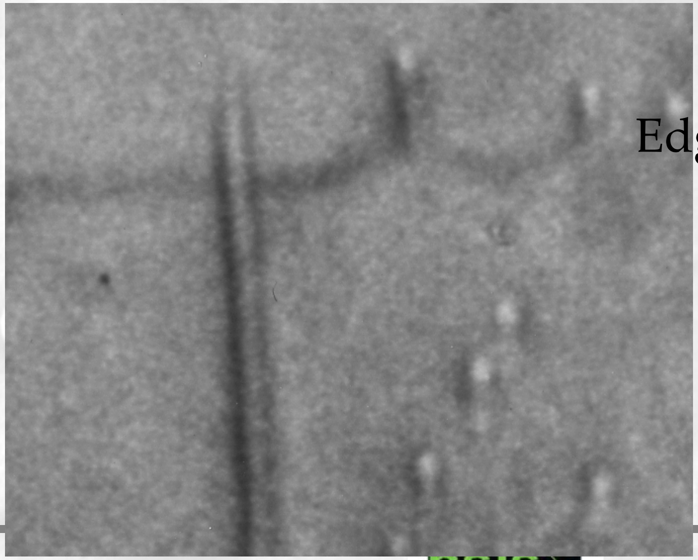
Comparison between white beam and monochromatic

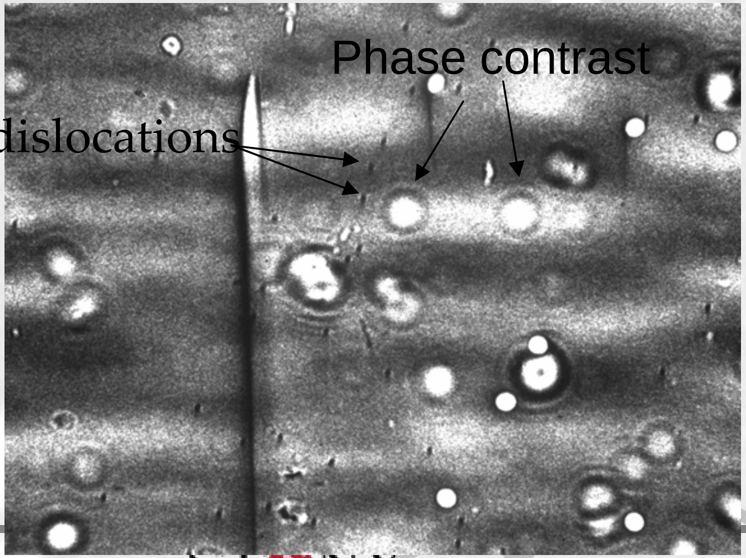
I



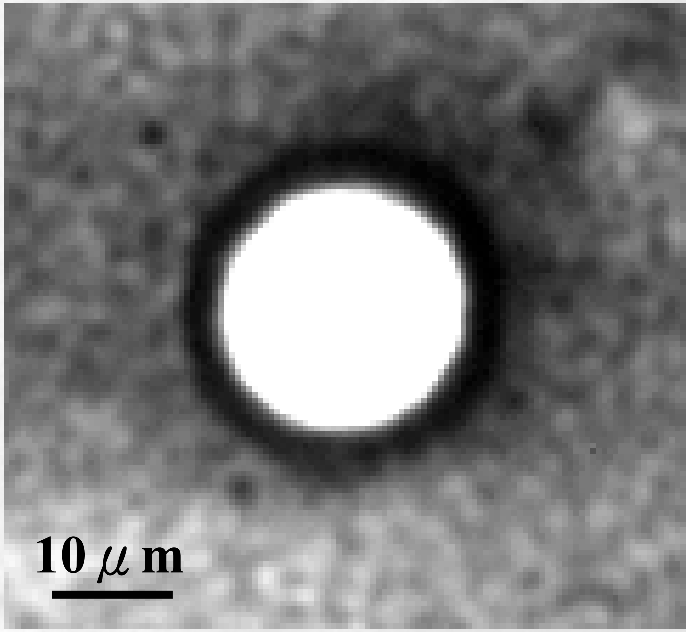


II

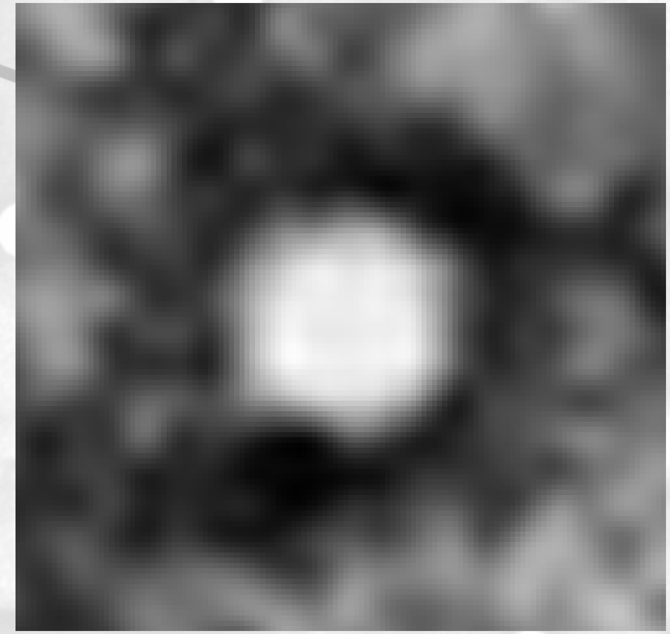




X-ray topographic image of elementary screw dislocations in SiC



Monochromatic Topo at APS

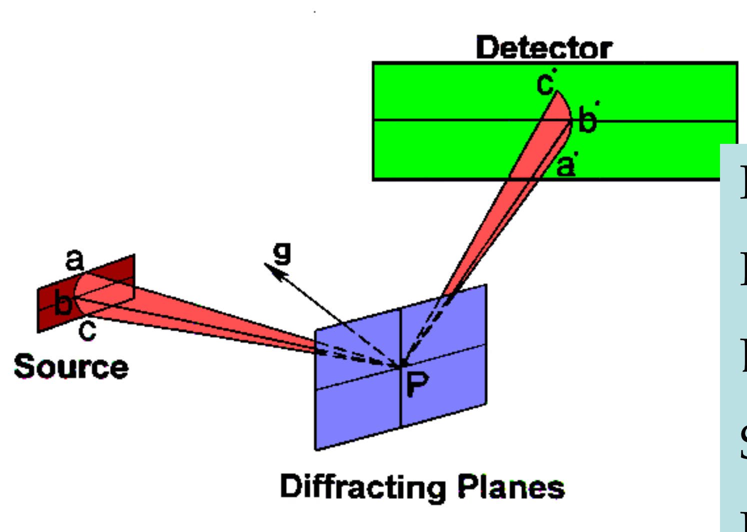


White beam Topo at NSLS

Burgers vector: 10Å

- At D_{sf}=12.5 cm, the diameter of the recorded 1c TSD image is ~19.5 μm in excellent agreement with ray-tracing simulation.
- For closed-core TSDs or MPs, Burgers vector magnitudes can be readily determined based on knowledge of image diameter as a function of D_{sf}.

Theoretical resolution of x-ray topography



$$R = a'c' = \frac{(ac)(Pb')}{bP}$$

First-generation:

NINA: R= 500 μm × 10 cm / 47 m =1.06 μm

LURE: R=1500 μm × 10 cm / 20 m =7.5 μm

Second generation - NSLS (X19C):

R= 100 μm × 10 cm / 25 m =0.4 μm

Third-generation –

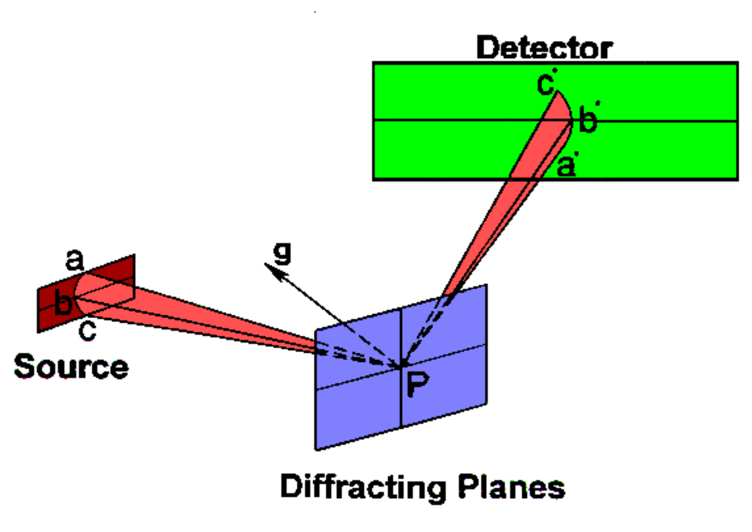
APS (X33-BM): R= 30 μm × 10 cm / 50 m =0.06 μm

ESRF (ID-19): R= 30 μm × 10 cm / 145 m =0.02 μm

Theoretical resolution at X33-BM APS and ID-19 ESRF is one order higher than X19C NSLS.

NSLS-II: ????

Source-to-specimen distance D_{ss}



$$R = a'c' = \frac{(ac)(Pb')}{bP}$$

At NSLS: D_{ss}=25 m

At APS: D_{ss}=50 m

At ESRF: D_{ss}=145 m

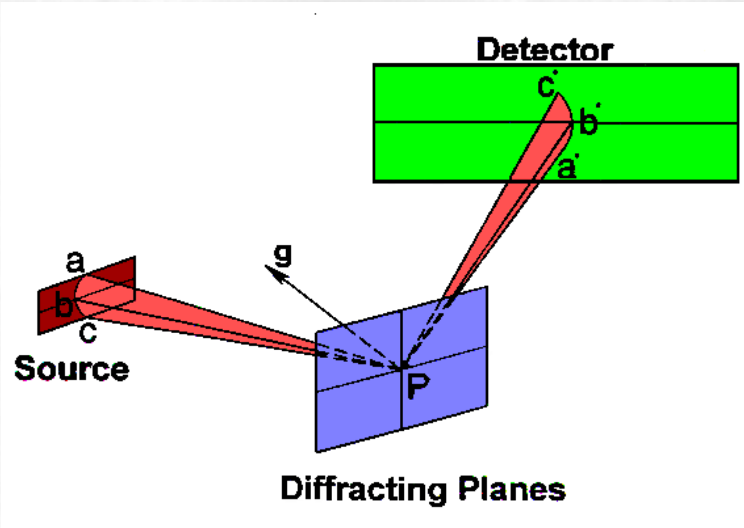
At NSLS-II: ??

Assume D_{sf}=10 cm and a source size 100 μm:

D_{ss}	100 m	ESRF: 145m	500 m	1000 m
Resolution	100 nm	~70 nm	20 nm	10 nm

10 nm: comparable to regular TEM?

Source size



$$R = a'c' = \frac{(ac)(Pb')}{bP}$$

At NSLS: 100 – 140 μm

At APS: 30 μm

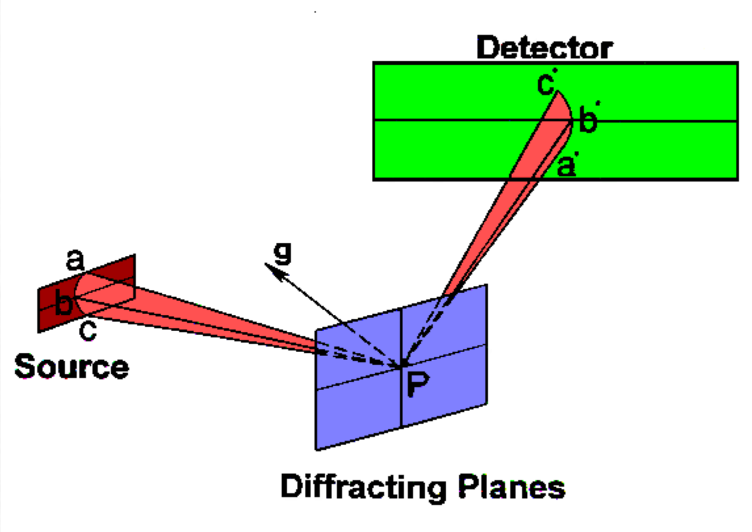
At ESRF: 30 μm

At NSLS-II: ??

Assume D_{sf}=10 cm, D_{ss}=1000 m and a source size 10 μm:

Resolution = 1 nm !

Specimen-to-film distance D_{sf}



$$R = a'c' = \frac{(ac)(Pb')}{bP}$$

In white beam, the D_{sf} is limited because of the strong background due to the diffuse scattering.

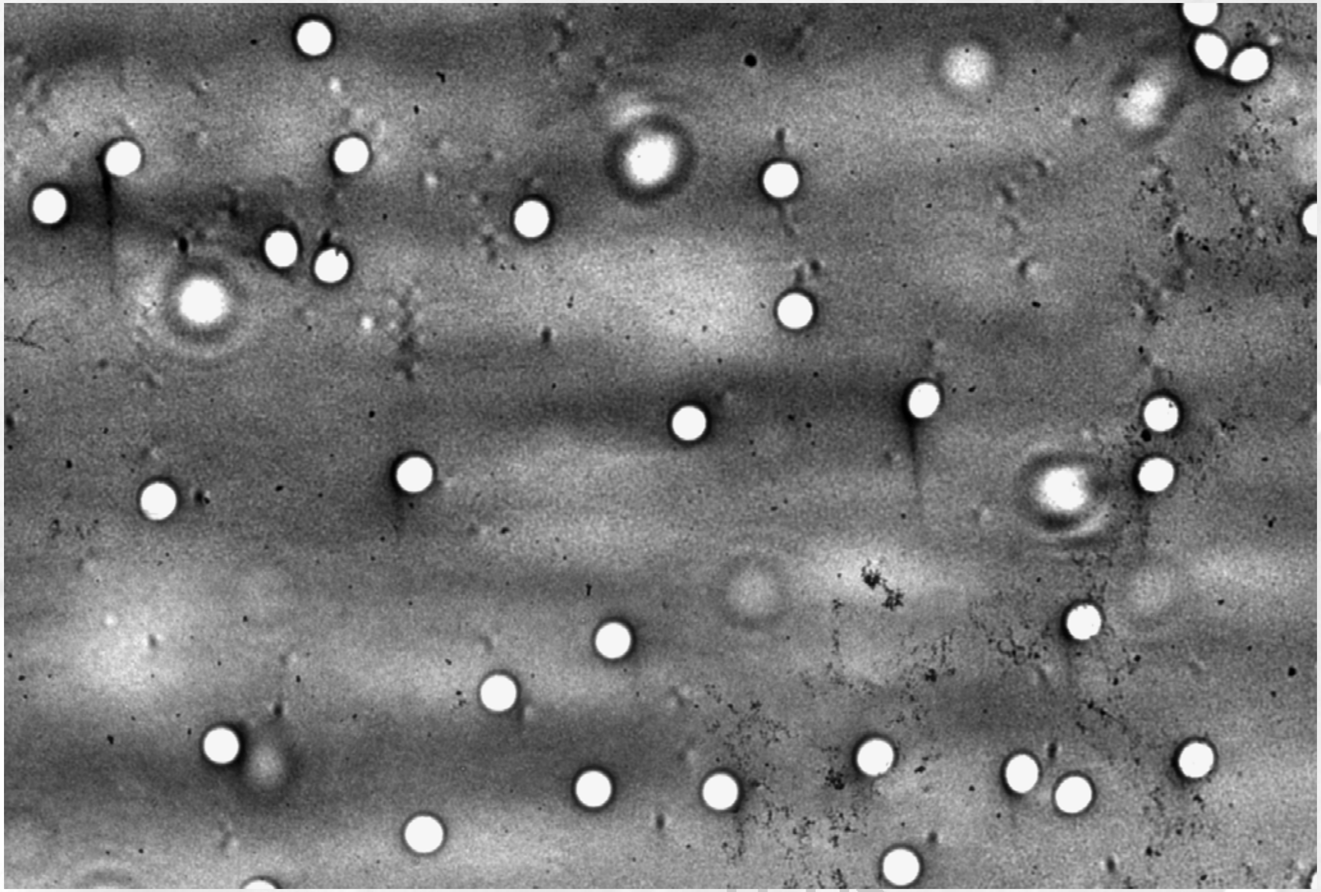
However, using monochromatic beam, D_{sf} can go below 1 cm if appropriate geometry is used (e.g., grazing-incidence geometry).

The resolution can be as low as 1 nm/cm.

<u>1 nm: comparable to high-resolution TEM?</u>

The actual resolution is limited by the image width of the dislocations

Ray-tracing simulation – a simple way to interpret topographs



White circles: topographic images of elementary screw dislocations in SiC (Burgers vector magnitude 10 Å)
Small black dots: edge dislocation (Burgers vector 3.08 Å)

Simulation of defects in SiC

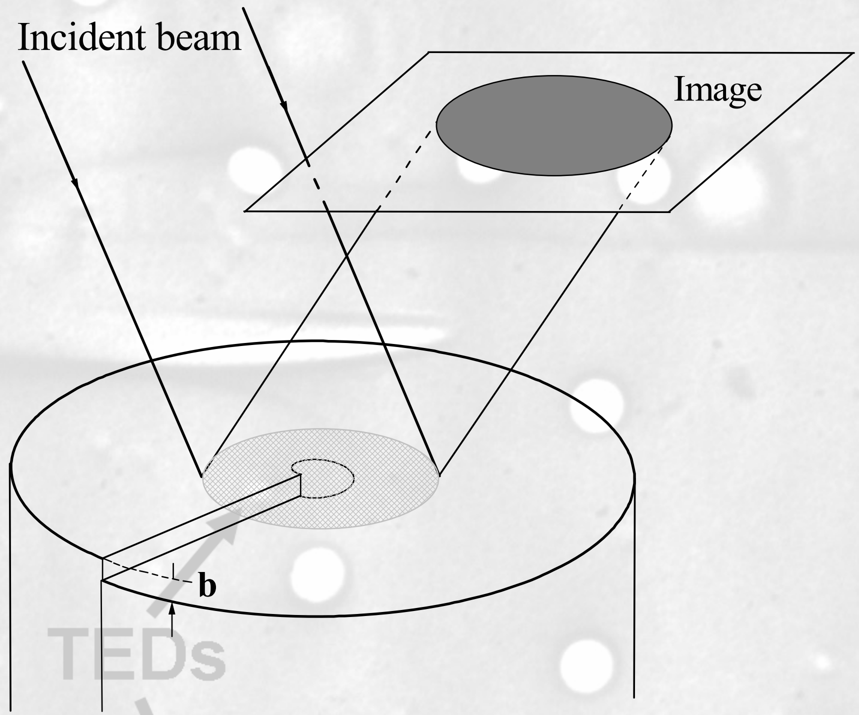
Back-reflection images are much larger than the hollow pipe diameters.

Micropipe diameters 0.1 ~ 4 µm, image size 18 ~ hundreds of µm.

How are the micropipe/dislocation images formed? Current theory inadequate.

Conventional understanding of dislocation contrast on X-ray topography

- Near the dislocation core, crystal lattice is highly distorted, kinematic diffraction mechanism dominates.
- Far away from the core, little distortion, dynamical diffraction mechanism dominates.
- Kinematic diffraction intensity is much stronger than dynamic intensity.
- Dislocation images should appear as **black disc**



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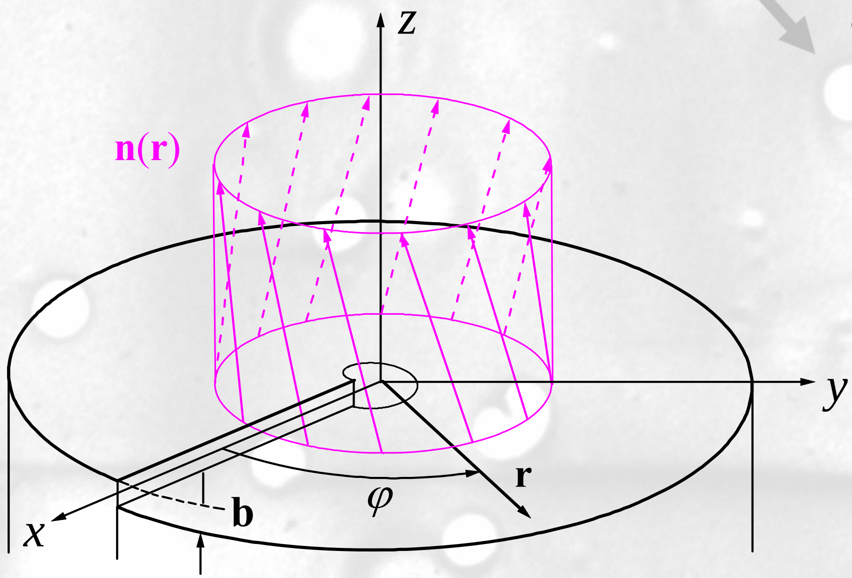
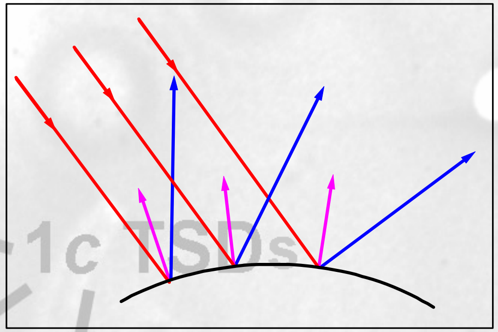
Continuous distortion of basal lattice plane due to screw dislocation



- Lattice displacement due arising from screw dislocation, normal to free surface:

$$\mathbf{u} = \begin{cases} u_z = \dfrac{b\varphi}{2\pi} & \textit{(Fundamental equation)} \\ u_r = 0 & \\ u_\varphi = -\dfrac{br}{2\pi(\sqrt{r^2+z^2}-z)} & \end{cases}$$

(Surface relaxation arising from image force)

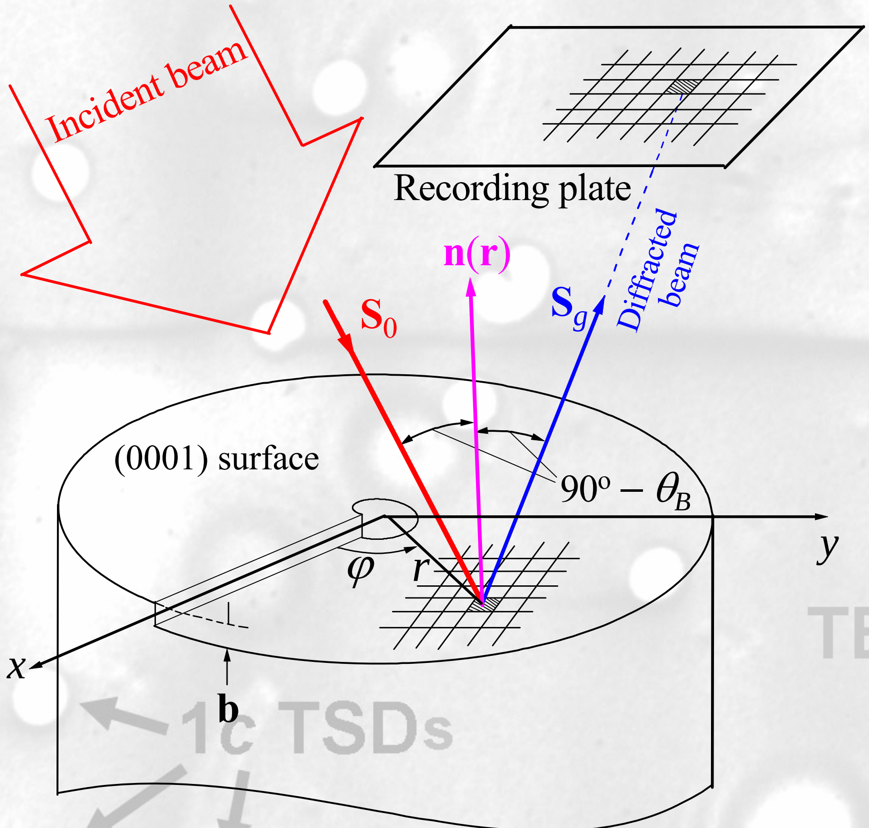


- Local normal of the diffracting plane:

$$\mathbf{n} = \begin{Bmatrix} n_z \\ n_r \\ n_\varphi \end{Bmatrix} = \begin{Bmatrix} -\dfrac{2\pi r}{(\sqrt{b^2+4\pi^2 r^2}} \\ 0 \\ -\dfrac{b}{(\sqrt{b^2+4\pi^2 r^2}} \end{Bmatrix}$$

Simulation of screw dislocation contrast

Kinematic theory — Ray-tracing method ⇒ Direct image



- White-beam diffraction equations:

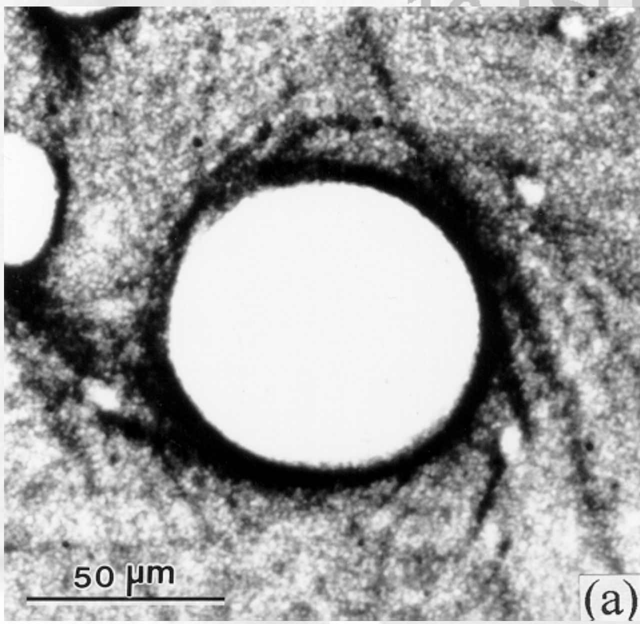
$$\mathbf{s}_g = \mathbf{s}_0 + 2\sin\theta_B \mathbf{n}$$

$$\theta_B = \mathbf{90^o} - \cos^{-1}(-\mathbf{s}_0, \mathbf{n})$$

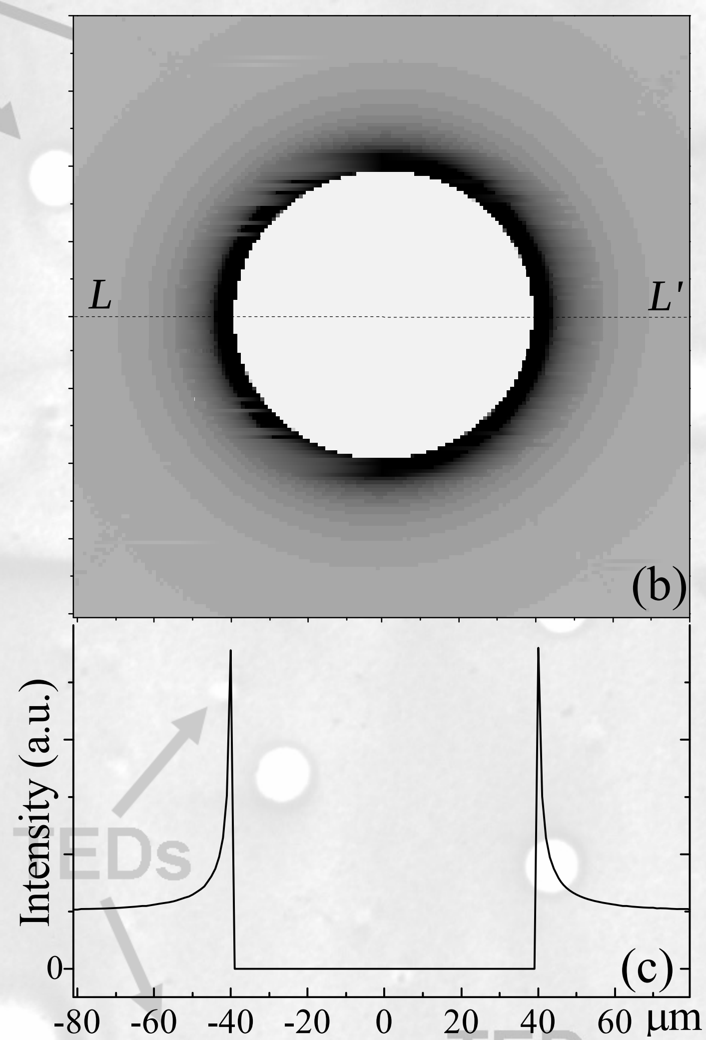
$\mathbf{s}_0$ — constant incidence direction
$\mathbf{s}_g$ — *locally* diffracted beam direction
θ_B— *local* Bragg angle

$\mathbf{n}$, $\mathbf{s}_g$, θ_B are all functions of position vector $\mathbf{r}$

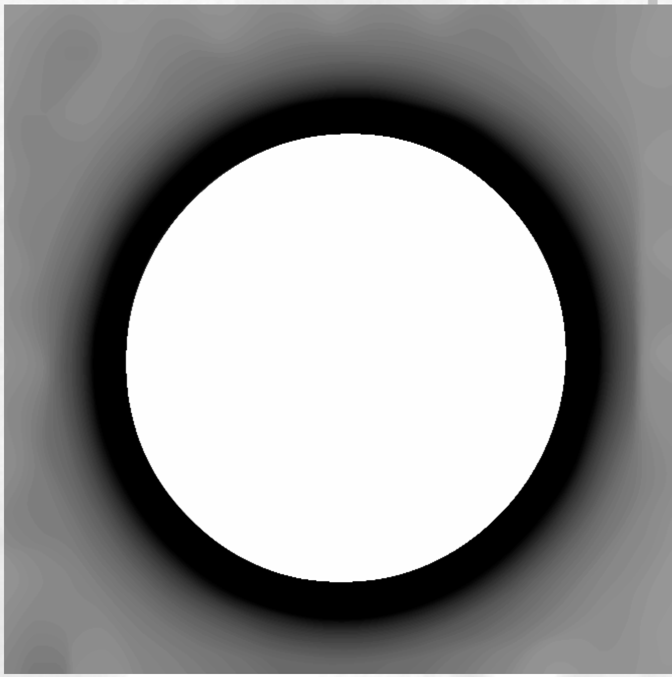
Simulation of an 8c micropipe image



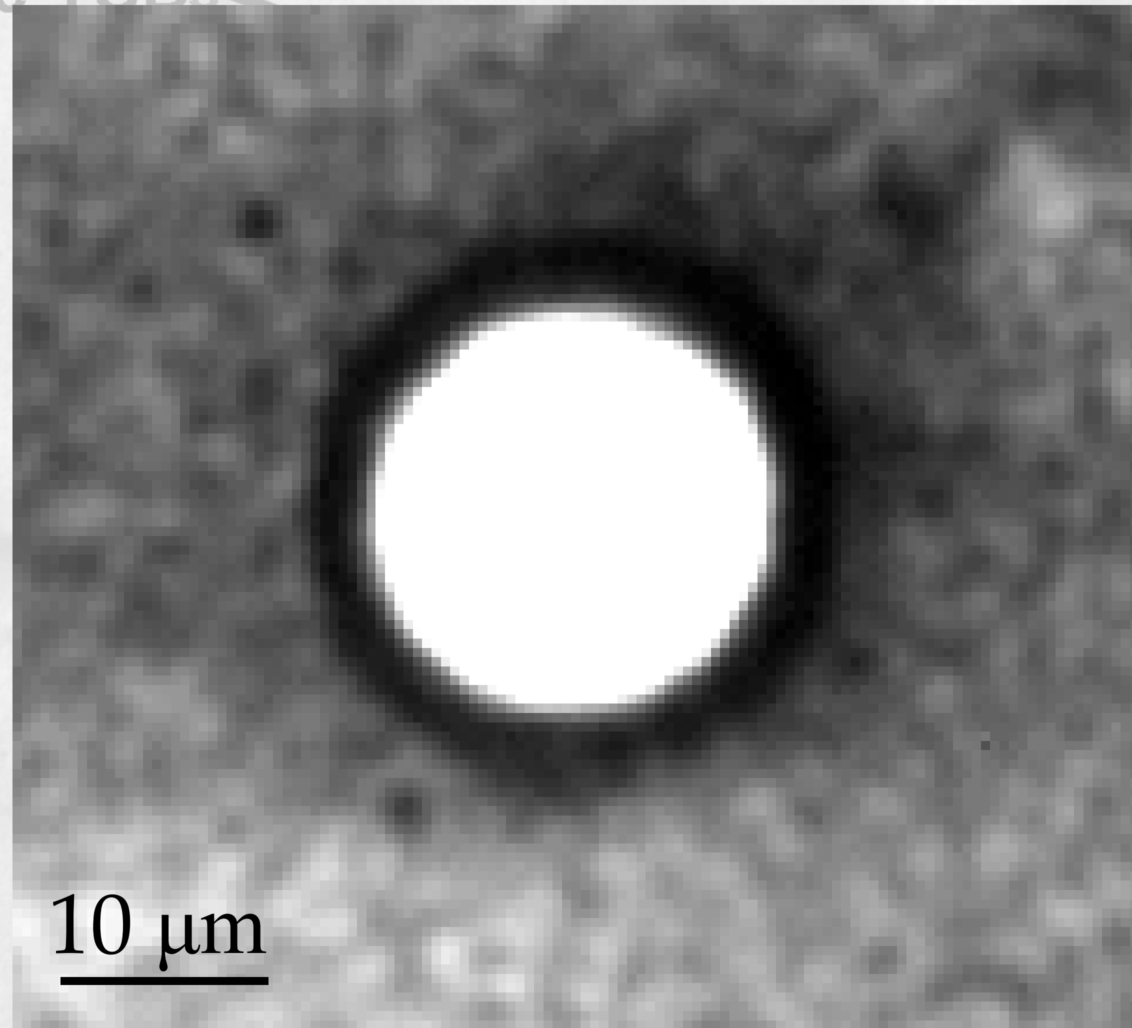
Recorded image of a micropipe



Simulation of an 1c screw dislocation image

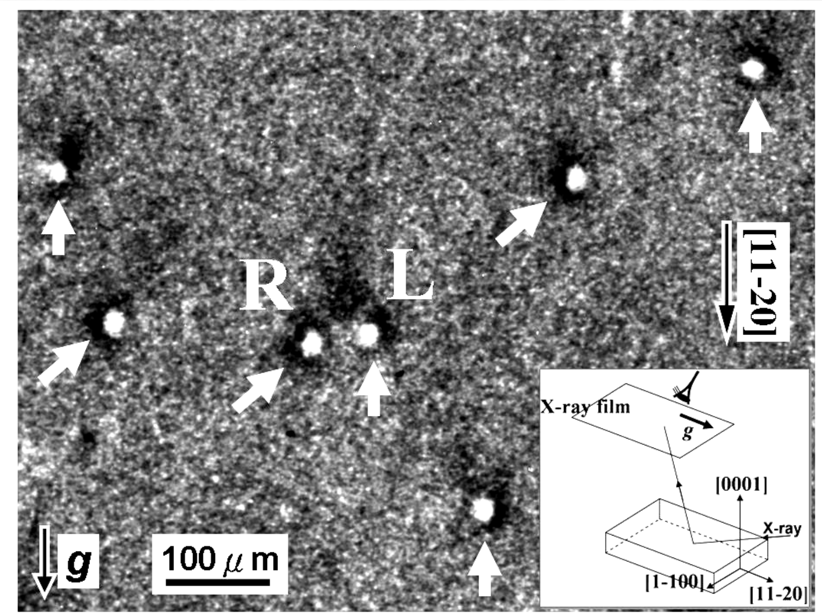


Ray-tracing simulation of 1*c* TSD

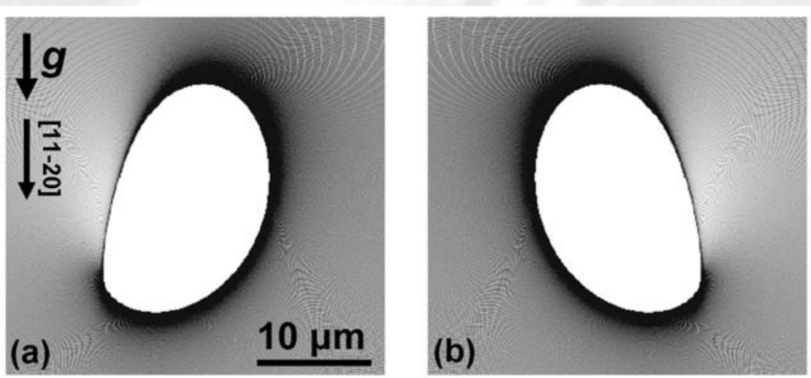


Monochromatic Topo at APS

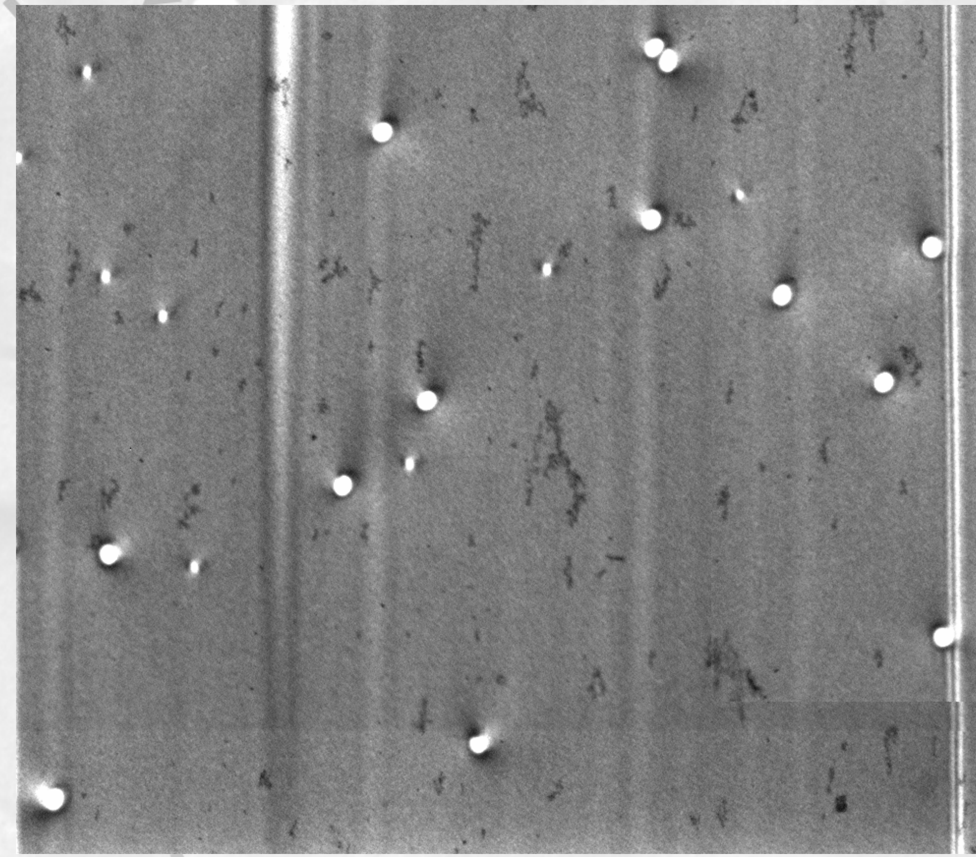
Reveal the dislocation sense of 1c screw dislocations



White beam

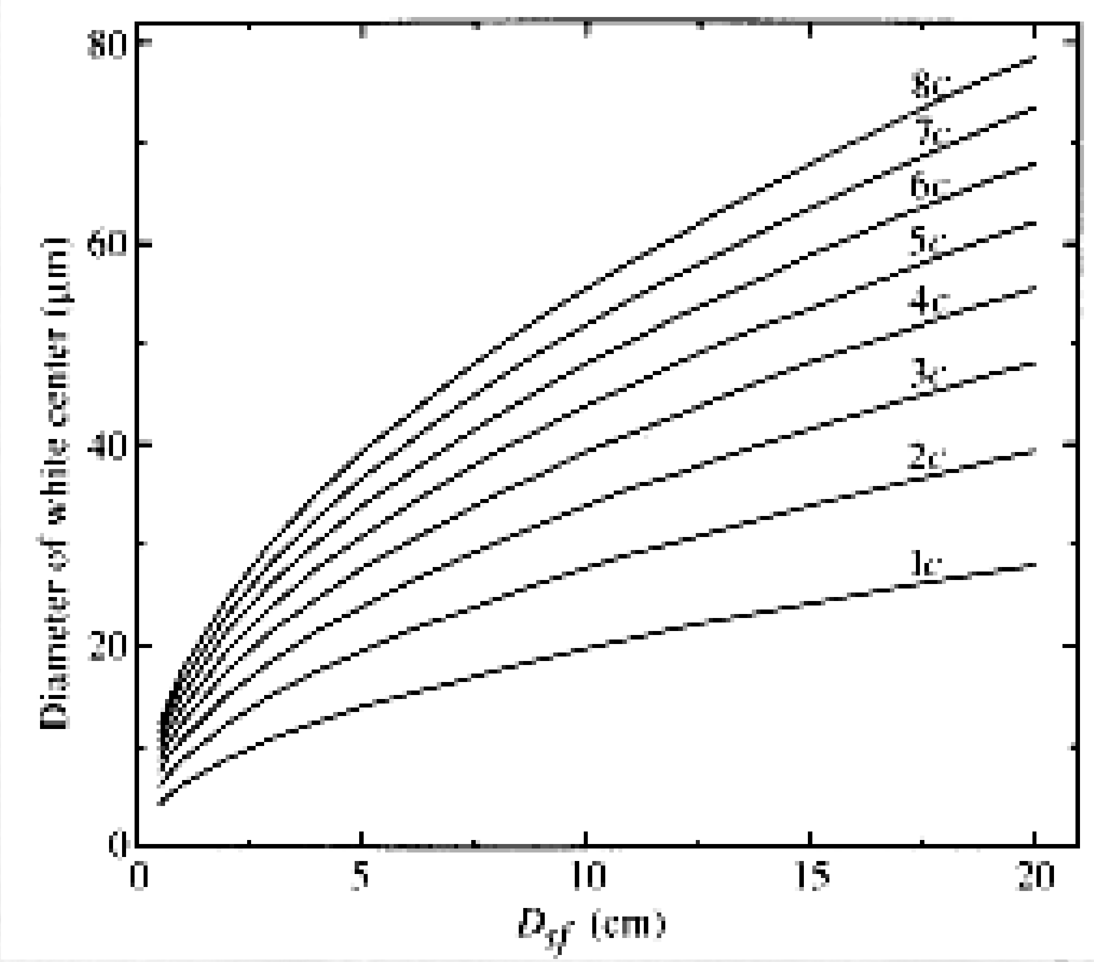


simulation



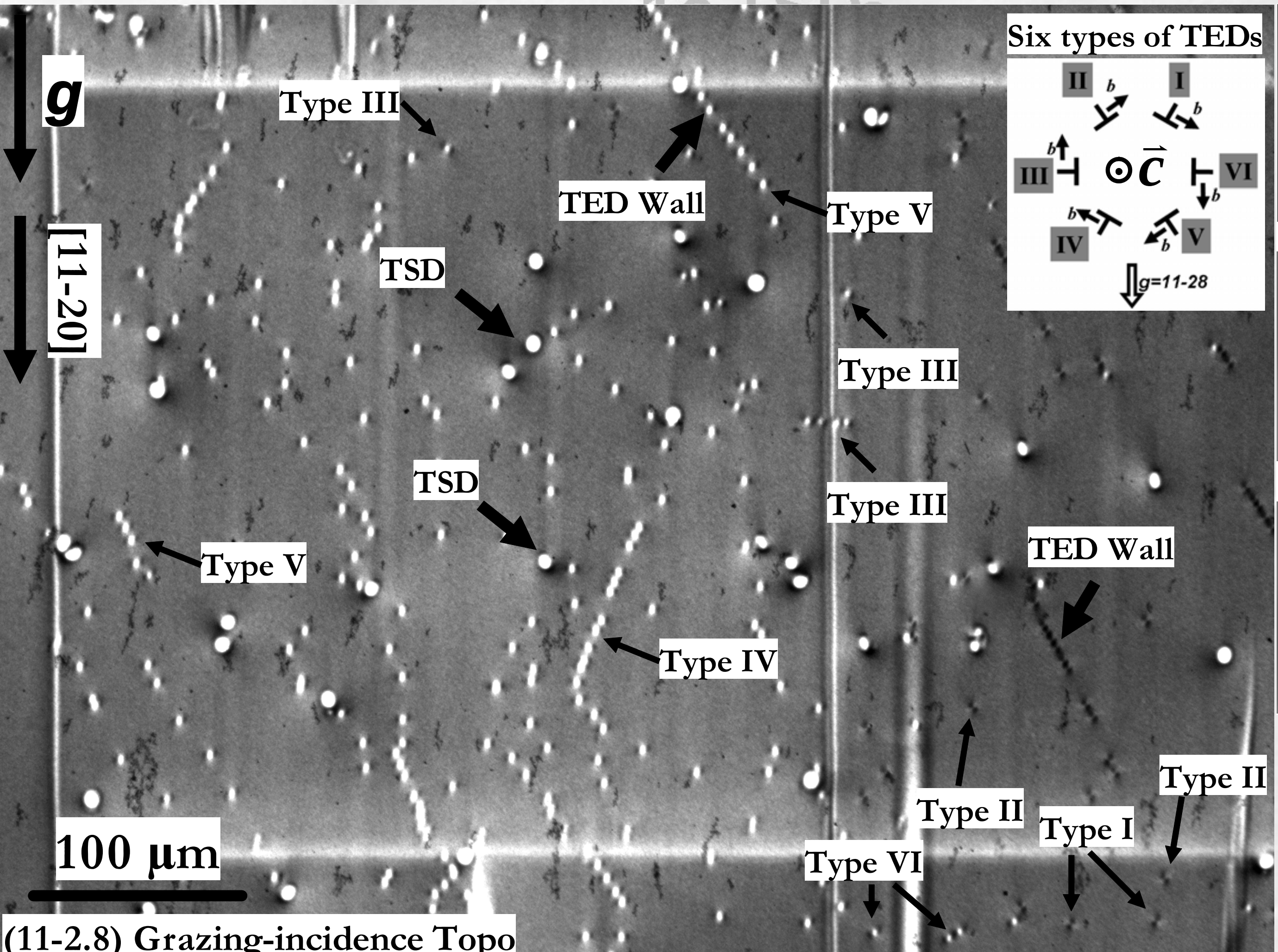
mono

Image size of screw dislocations in SiC vs. D_{sf}

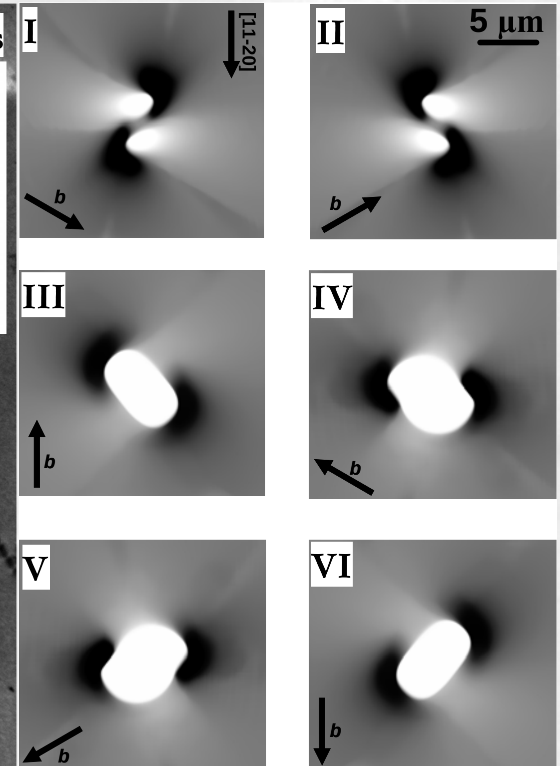


- dislocation image size reduces as D_{sf} decreases
- the image size of threading elementary screw dislocations is ~5 μm at D_{sf}=1 cm
- image size of threading edge dislocations is ~2 μm at Dsf=1 cm

Threading edge dislocations



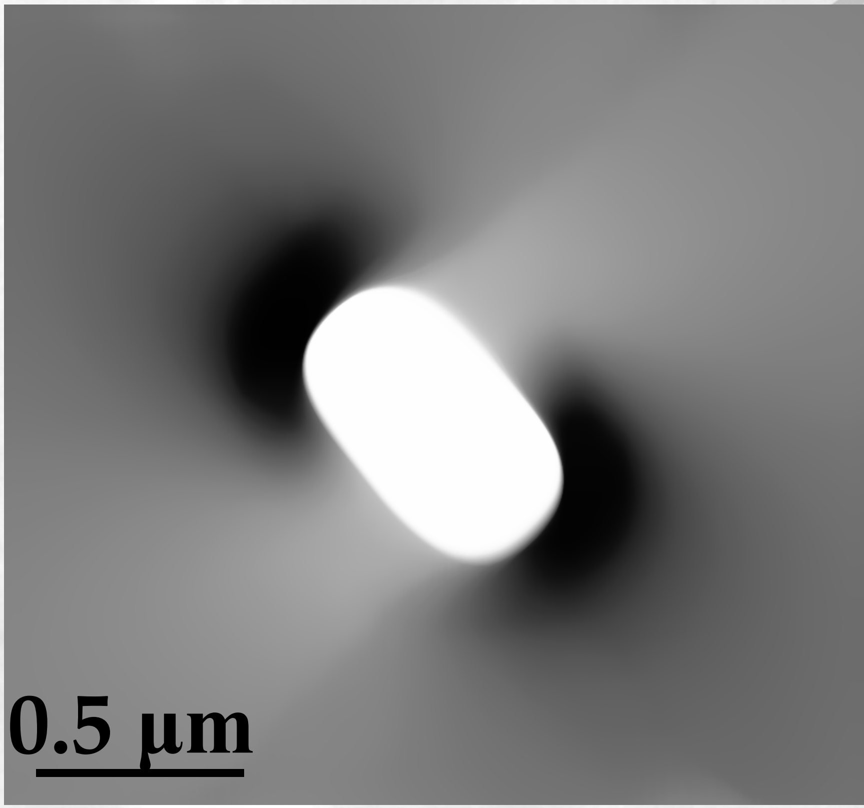
(11-2.8) Grazing-incidence Topo



Ray-tracing simulation

D_{sf}=10cm

Images size at reduced D_{sf}

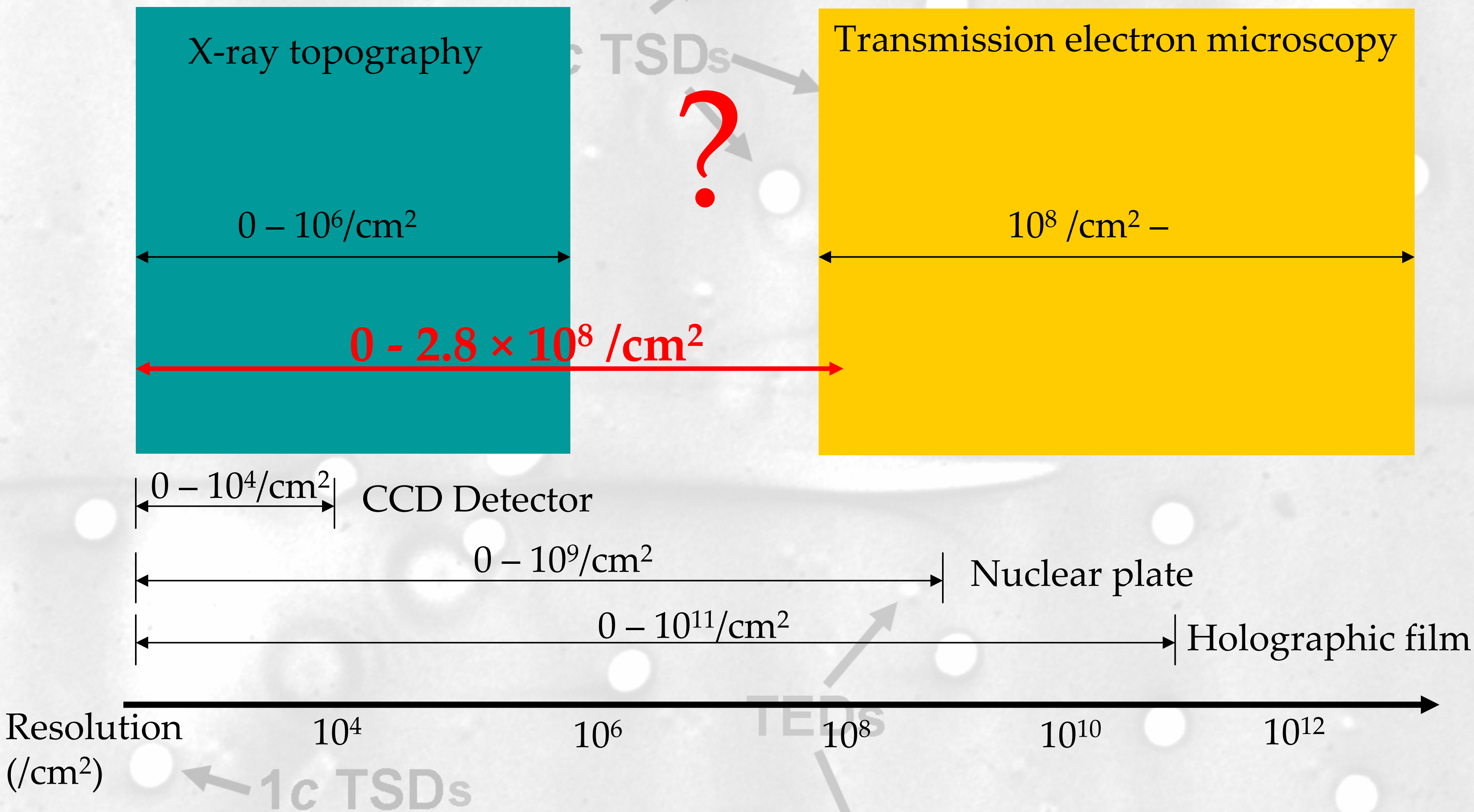


Edge dislocations in SiC have typical magnitude of Burgers vectors ~3 Ǻ.

Simulated edge dislocation image at D_{sf}=0.1 cm.

- Dislocation image width at D_{sf}= 0.1 cm is approximately 0.6 µm.
- The maximum observable dislocation density is **2.8×10^8 /cm²** !

Fill in the resolution gap between TEM and XRT



The resolution gap between XRT and TEM can be filled!

Improvement of strain/stress measurement at NSLS-II

At NSLS-II:

▪ The sharpness of reticulography can be improved by large source-to-specimen distance and small source size. Higher strain sensitivity.

▪ Finer-scale mesh can be used in x-ray reticulography. Therefore, higher spatial resolution can be achieved.

Proposed suites of beamlines:

- Interchangeable Monochromatic/white beam (former downstream from the latter)

Beamline specifications:

- source: Damping Wiggler?

-2T, 3-Pole Wiggler?

- optics: Asymmetric geometry monochromator (to spread beam)

- world leading endstation: large D_{ss}?

Conclusions

▪ The resolution of XRT is determined by the source size, source-to-specimen distance and specimen-to-film distance. A resolution of 1 nm can potentially be achieved at NSLS-II.

▪ The maximum resolvable dislocation density in XRT is limited by the dislocation image width. Experimental and simulated results indicate that by reducing the specimen-to-film distance, the image dimension of a typical dislocation can be as small as 0.6 μm. An actual maximum resolvable dislocation density of $10^8/\text{cm}^2$ can be achieved enabling the gap in dislocation resolving power between XRT and TEM to be filled.

▪ Higher strain sensitivity and spatial resolution for strain/stress measurement can be achieved at NSLS-II.